

Exhibit B

FAX TRANSMITTAL

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To

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This fax includes 17 pages including this front page.

You are always taking care of it.

I am remiss in writing very much and I am truly sorry about that.

Although it became very late, I am sending you a draft document for a patent by fax.

The reason of the delay is in that our company has a plan to obtain a broad scope of patent including product process claims such as Toshiba's Ti target patent which is enclosed herewith.

It is said by Tg manufactures that the enclosed Toshiba's patent is a highly evaluated important patent.

So please refer to this patent for your information. It would be fortunate if you instruct us once again for details, after you examined this matter.

Please receive it. I request it with the feeling of gratitude.

A: Claims

1. An Ag alloy sputtering target material with 0.1 to 10% by volume each of one or a plurality of elements selected from the group consisting of Au, Pd, Al, Cu, Ta, Ti, Rh, and Cr added.

2. An optical recording medium using the target material according to claim 1 for forming a reflection film.

3. An optical recording medium using an AgPd alloy material with a 0.5 to 3% by volume Pd composition as a reflection film, and a sputtering target material for reflection film formation.

4. An optical recording medium using an AgPd alloy material with a 0.1 to 3% by volume each of one or a plurality of elements selected from the group consisting of Au, Al, Cu, Ta, Ti, Rh, and Cr added, and with a 0.5 to 3% by volume Pd composition as a reflection film, and a sputtering target material for reflection film formation.

5. An Ag alloy sputtering target material with a rich weather resistance with respect to a non-metal element such as oxygen, sulfur, and chlorine.

Detailed Description of the Invention

[Object of the Invention]

(Industrial Field of Application)

The present invention relates to an Ag alloy sputtering

target material for reflection film formation, having a stably high reflection ratio of 90% or more as a reflection film to be used in an optical recording medium in the case of a less than 780 nm wavelength, useful as a material having a high durability with respect to the pollution of a non-metal such as oxygen, sulfur, and chlorine compared with the Ag, and an optical recording medium using the same.

(Prior Art and its Problems)

As a reflection film material for optical discs such as a CD, and a DVD, an Al, or an Al alloy is commonly used. The above-mentioned material is used also for optical magnetic discs such as an MD, and MO, or rewritable optical recording media such as a phase change type optical disc.

This is because useful improvements that a certain reflection ratio or more can be provided in a specific optical wavelength range for reproducing the optical recording media as well as stable heat conduction characteristic and gap covering property with respect to a groove pitch produced in a medium itself can be provided, that a rich weather resistance with respect to the pollution derived from a non-metal contained in the air can be provided as a medium merchandise so that the aging change is extremely small over a long term, that a rate extremely high in sputtering can be provided with respect to the Al in a sputtering method often used as a method for forming a reflection film, or the like, are confirmed.

However, the reflection ratio of the Al, or the Al alloy is about 85% in, for example, a 780 nm range, and thus a sufficiently high reflection ratio cannot be provided, depending upon the application.

In the CD-R, an Au has been adopted as a reflection film material conventionally for the reason that a sufficiently high reflection ratio cannot be obtained by an Al-based material. However, since the Au involves a problem of the high cost, as a substitute material for the Au, an Ag, or a Cu is examined and adopted in the present situation. Although the Ag is the most expected material, since it is chemically active with respect to elements and ions such as the chlorine contained in the sea water, and oxygen or sulfur in the atmosphere so that many problems are involved in terms of the weather resistance in a special environment, it is not a material having characteristics satisfactory as the Au substitute.

Japanese Patent Laid-Open Application (JP-A) Nos. 57-186244, 7-3363, and 9-156224 report improvement of the weather resistance of the Ag by adding a specific impurity. An AgCu alloy (Ag > 40% by volume), an AgMg alloy (Mg: 1 to 10% by volume), AgO:M (M = Sb, Pd, Pt) alloy (O: 10 to 40% by volume, M: 0.1 to 10% by volume) are described, respectively. However, as to these materials, the composition ranges are wide, and the relationship between the weather resistance and the reflection ratio is not explicit. In particular, improvement of the

weather resistance with respect to addition of a slight amount of an impurity seems not be sufficient. Reliability in adopting in an optical recording medium is not explicit as an effect to be provided by the addition, and thus the usefulness as a material is unclear in many aspects.

Moreover, there are many vague points without clear data in terms of whether or not a thin film can be produced easily by a sputtering method, production easiness of a useful Ag alloy target when it is used as the target material, the relative evaluation of the difference in the film formation rate in the process with respect to the Ag in the actual film formation, or the like. Besides, as to the Mg, there are many difficult aspects to be discussed before use because it is classified in the alkaline earth metal and extremely chemically unstable for the material physical property imparted to the element or the ion, it cannot satisfy the demand for the weather resistance improvement with respect to the chlorine, or the like.

[Configuration of the Invention]

(Problems and Means for Solving the Problems, and Effects)

In order to achieve the above-mentioned object, the present inventors had elaborately discussed on maintenance of a high reflection ratio compared with the Ag, improvement of the weather resistance, easiness of the production method in the case of an alloy, and stability and convenience of the sputtering process in the use as a target. As a result, the

alloy comprising two elements with the Pd as the first adding element disclosed in the claim, and the alloy with the AgPd alloy comprising the two elements as the base material and a second adding element added thereto were discussed, and the experiment and evaluation were executed.

The first reason why the Pd was selected is that the material satisfy the conditions for easy production, such as capability of restraining segregation of the adding element Pd with respect to the entire alloy during the melting process for producing the alloy and after cooling and solidification owing to the small specific gravity difference between 10,491 g/cm³ of the Ag and 12,02 g/cm³ of the Pd, and absence of the risk of the inter-metal compound formation in each process for the production. Other reasons are as follows.

As mentioned above, since the main material Ag is easily bonded with the sulfur, in the case it is left in the atmosphere for a long time as a solid or a thin film, the interface in contact with the atmosphere is fiercely reacted with the sulfur so as to become a silver sulfide (Ag₂S) with a black color, and thus the reflection characteristic is lost. Besides, it is fiercely reacted also with the chlorine so as to become a silver chloride (AgCl), and thus the surface reacted part becomes opaque. Furthermore, the part reacted with the chlorine grows and spreads so as to loose the reflection characteristic and the physical property as the Ag. However, it is relatively stable

with respect to the oxygen and the hydrogen. In particular, it is highly stable with respect to the hydrogen so that there is no risk of bonding with the oxygen after leaving for a long time in an oxygen atmosphere or bonding with the hydrogen after and leaving in the state soaked in the water (H_2O), it is frequently used as an additive material for a photosensitive material or a high melting point wax material for the sake of the barrier property with respect to the oxygen or the hydrogen.

In contrast, the Pd is durable with respect to the reaction with the sulfur and the chlorine unless it is reached at a high temperature, and thus it is well known as a material chemically highly stable with respect to the chlorine and the sulfur. However, since it has a characteristic of adsorbing and activating the hydrogen, in the case of producing a plate material by a melting method, a slight amount of a Ti is added often as the barrier material with respect to the hydrogen.

Based on the above-mentioned reasons, by adding a predetermined amount of the Pd to the Ag so as to homogeneously dispersing the Pd in the Ag grain boundary, according to the interaction of the hydrogen resistance and the oxygen resistance of the Ag, and the chlorine resistance and the sulfur resistance of the Pd, high improvement of the weather resistance can be realized with respect to the pollution by a non-metal element in the atmosphere of the chlorine, the oxygen, the hydrogen, or the sulfur, or in a special environment, or in the environment or

the atmosphere required at the time of being adopted in an optical recording medium compared with the Ag.

The experiment content according thereto is as follows.

"Experiment content is as disclosed in the proposal draft by the Chief Researcher Mr. Aratani of Sony Corp. Gigabite Research Institution."

Composition of the Experiment Specimens

Ag-Pd (Pd addition amount = 0.3% by volume, 1% by volume, 2.5% by volume, 5% by volume)

According to the experiment result with the above-mentioned specification, improvement and realization of the weather resistance was confirmed. Moreover, dependency on the weather resistance of the Pd composition was confirmed. In comparison with the case of the Ag, it was confirmed that the two element alloy with the Pd added has the reflection ratio lowered inversely proportional to the addition amount. According to the addition amount, in a wavelength range of less than 780 nm, it is lowered by about 1 to 15%. However, as to those having a small addition amount, compared with the case of Al or an Al alloy, the reflection ratio is higher by more than 5 to 10% in the same wavelength range, and thus the superiority to the Al based material can sufficiently be observed. However, in consideration as a substitute of the Ag or the Au, although the reflection ratio can be improved to some extent by the Pd addition amount, a slight deterioration cannot be avoided.

Then, for restraining the decline of the reflection ratio with the weather resistance improved by adding the Pd to the Ag, addition of a second adding element was discussed, and the experiment and evaluation were executed.

Here, whether or not improvement of the reflection ratio can be realized by adding an element having a certain reflection characteristic or more such as the Au, Al, Cu, Ta, Ti, Rh, Cr, or the like as disclosed in claim A-4) was evaluated by an experiment with the same condition as the above-mentioned case of the AgPd.

"Experiment Result"

From the experiment, in comparison with the Ag, even in the same wavelength range, decline of the reflection ratio was restrained to about 2 to 3% at the maximum. Furthermore, as to the improvement of the weather resistance, which is the characteristic mostly required, it was maintained at the state improved with respect to the case of the Ag without ruining.

The composition of the Ag-Pd-X (X = Au, Al, Cu, Ta, Ti, Rh, or Cr) alloy (Pd: 0.1 to 3% by volume, X: 0.1 to 3% by volume) defers according to the optical recording medium, such as the CD-R, the optical magnetic type, the phase change type, or the like.

This is because the heat conduction ratio, the heat expansion ratio, the residual stress as a film, and the joint between the upper and lower part layers and the interface part,

the adherence state, or the reactivity differ drastically in the case of forming a layer as a film as the characteristics required as a reflection film other than the reflection ratio and the weather resistance, and thus the addition amount composition should be changed according to each required item.

In order to determine the composition ratio of the Ag and Pd, and the second adding element X, the following items were discussed as the items to be confirmed.

The most prominent characteristic of the Ag compared with the other metal elements is not only the reflection ratio in a specific wavelength range but also the best conductivity as the heat or the electricity, and the best characteristic in the case of actually melting for producing a target in terms of the processing property and the spreading property.

Moreover, as to the heat conduction ratio, it is extremely high compared with the Au, the Al, or the like as the subject to be substituted as the reflection film. It has a high value that the heat conduction ratio should be lowered in the case of substituting.

In the case of adopting as a thin film, the easiest means for combining these characteristics with a substitute subject material is considered to be adjustment by the film thickness in the case of forming a thin film. However, since each optical recording medium as a standard thickness as a product, it is difficult to have an extremely thin film

thickness. Therefore, in a sense, in addition to having a certain thin thickness, adjustment should be made to the substitute subject material such as the Au and the Al by increasing an adding amount of a material, setting an element with a low heat conduction characteristic as the additive.

Furthermore, since the Ag has the highest sputtering ratio among the metal elements as well as an extremely high sputtering rate in the process, it can be regarded as a material with the extremely high superiority in the production process with an extremely large amount of production is required such as an optical recording medium.

Also in this regard, it is considered that since the productivity improvement can be achieved by the high usefulness of the Ag, the material physical property itself should not be ruined.

Therefore, an experiment was executed for confirming the adding amount capable of restraining the decline of the sputtering rate by observing fluctuation of the sputtering rate depending on the adding element and the amount thereof.

"Experiment Method"

An Ag-2% by volume alloy was produced in a (76.2 mm (6 mmt shape, and a second adding element was produced as a pure metal each in the same shape.

Each of the produced metals was installed in a sputtering device. By the electric discharge of an AgPd alloy

to be the base material and a metal to be the second adding element at the same time for forming a three element alloy film.

At the time, for the relative evaluation, a film was formed only for the Ag and the AgPd alloy for measuring data to be the reference of the rate difference.

A film was produced by the RF magnetron sputtering method with a 1,000Å thickness for the film to be produced. The second adding element to have the electric discharge together with the AgPd alloy was 3% by volume at the maximum as in the claims. Also all the sputtering conditions other than the electric power amount with different application conditions for the electric discharge were provided same for executing the relative evaluation in the same conditions as much as possible.

The sputtering conditions include a 4×10^{-3} reaching pressure, a 0.76 Pa sputtering pressure, and a 20 sccm gas flow amount as an Ar atmosphere using an Ar gas as the sputtering gas and the atmosphere.

"Experiment Result"

| Production material for the alloy film | Film formation electric power (ratio) | film formation time |
|---|---|---------------------|
| Ag | 500W | 46sec |
| Al | 500W | 152sec |
| Au | 500W | 122sec |
| Ag-Pd | 500W | 55sec |
| Ag-Pd-Cu | 500W:45W | 60sec |
| Ag-Pd-Ti | 500W:215W | 49sec |
| Ag-Pd-Ta | 500W:35W | 60sec |
| Ag-Pd-Rh | 500W:45W | 58sec |
| Ag-Pd-Cr | 500W:55w | 57sec |
| Ag-Pd-Al | 325W:500W | 84sec |
| Ag-Pd-Au | 500W:5.2W | 60sec |

From the above-mentioned experiment result, the film formation time increase by about 10 to 50% was confirmed compared with the Ag, but in comparison to the Au and the Al as the subject for substitution, it was cut by half or more. It was confirmed that even in the case of 3% by volume of the adding amount of a second adding element as the maximum value in the claims, the superiority of the Ag sputtering rate with respect to the other alloys is not ruined as well as the maximum time to be increased actually.

Next, easiness of the alloy production method and the dependency on the composition were to be confirmed. As to the

shape to be produced, it is same as the above-mentioned experiment, that is, a $\phi 76.2$ mm x 6 mm shape.

In the case of producing an Ag alloy according to the claims, two production methods were adopted. As easy production methods, two methods including a dissolution method in an atmosphere same as the case of the Ag, and a melting method in a vacuum were executed.

First, in the case of producing the Ag alloy by melting, since the method of producing a base alloy, and thereafter adding the Ag to the base alloy and spreading the base alloy to the Ag standard amount seems to be easiest, a base alloy casting method is discussed.

The melting method in the atmosphere is as follows.

1) A base alloy is produced by melting and mixing an Ag-Pd-X alloy in an Ar atmosphere (400 to 600 torr) by an arc melting.

At the time, the melting operation is executed with a composition with 10 to 15% by volume of the Pd, and 15 to 20% by volume of the X (Cu, Ti, Ta, Rh, Cr, Al, Au) with the Ag a the absolute amount.

2) The Ag (the amount is obtained by subtracting the base alloy amount from the entire dissolving amount) is dissolved in a high frequency dissolving furnace. The melting temperature at the time is 1,000 to 1,500°C. As a crucible to be used for melting the Ag, a standard type graphite crucible

(for 0.1 to 0.2 liter) is used.

3) By adding an antioxidant after completely melting, solid solution with the oxygen during melting is restrained and prevented. As the antioxidant to be used at the time, borax, sodium borate, lithium borate, carbon, or the like, can be used.

4) After leaving for about 1 hour in the completely melted state, the base alloy of the above-mentioned item 1) is added. For further 0.5 to 1 hour, the melting operation is executed.

The melting temperature at the time is about 1,050 to 2,000°C.

5) The molten product is poured into an Fe casting mold with an alumina, or a magnesium-based talc applied on the inner surface.

For preventing shrinkage cavity, the Fe casting mold is heated preliminarily to about 300 to 500°C using an electric furnace, or the like.

6) When the cooling and solidification is finished in the casting mold, an ingot is taken out from the casting mold. It is further cooled to an ordinary temperature.

7) The pressed part at the ingot uppermost part is cut off and eliminated.

8) The ingot is rolled by a rolling machine so as to produce a 90 x 90 x 8.1 t plate.

9) A heat treatment is executed at 400 to 500°C in an

electric furnace with an Ar gas sealed, and kept for 1 to 1.5 hours. Thereafter, the plate material in the heated state is processed by a press machine, and left until it returns to an ordinary temperature for correcting warpage.

10) It is wire-cut into a product shape by a wire electric discharge cutting machine.

11) The product entire surface is abraded by a water proof abrasion paper for adjusting the surface roughness.

The melting method is a very standard production method. It is also a standard method as a method for producing a base alloy. In the case of adding and melting the Pd or other adding elements X to be discussed to the Ag, the production can be executed easily without the need of using a special method. Therefore, it is highly advantageous to adopt the Ag alloy in terms of both cost and production method.

As another standard method other than the above-mentioned, the graphite crucible can be replaced by a ceramic crucible made from an alumina, a magnesia, or the like, as the crucible for placing the melting material for the melting operation in a vacuum chamber. All the other conditions are same as the above-mentioned atmosphere dissolution.